



ATTORNEY'S DOCKET NO. M00925/70110 TJO

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

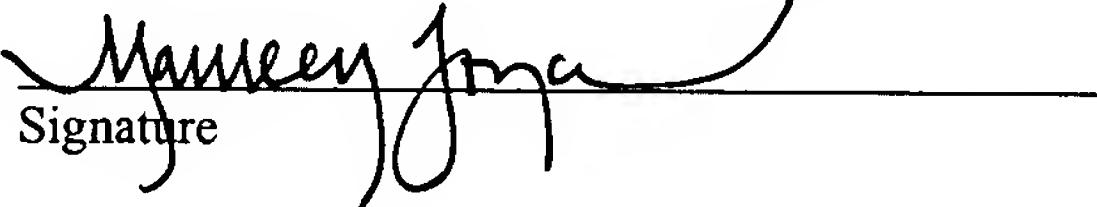
RECEIVED  
MAY 12 2003  
TC 1700

Applicant: Jackie Y. Ying et al.  
Serial No: 10/044,801  
Confirmation No.: 4734  
Filed: January 11, 2002  
For: NANOCRYSTALLINE APATITES AND COMPOSITES,  
PROSTHESES INCORPORATING THEM, AND METHOD FOR  
THEIR PRODUCTION  
Examiner: Carol M. Koslow  
Art Unit: 1755

---

**CERTIFICATE OF MAILING UNDER 37 C.F.R. §1.8(a)**

The undersigned hereby certifies that this document is being placed in the United States mail with first-class postage attached, addressed to Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the 5 day of May, 2003.

  
Signature

---

Commissioner For Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**MARKED-UP CLAIMS**

581. (New) A composition, comprising particulate apatite having an average apatite particle size of less than 1  $\mu$ m, an average apatite crystal size of less than 150 nm and a surface area of at least about 40  $\text{m}^2/\text{g}$ .

582. (New) The composition of claim 581, wherein the particulate apatite has a surface area of at least about 60  $\text{m}^2/\text{g}$ .

583. (New) The composition of claim 582, wherein the particulate apatite has a surface area of at least about 100  $\text{m}^2/\text{g}$ .

584. (New) The composition of claim 581, wherein the particulate apatite has an average apatite particle size of less than 0.5  $\mu$ m.

585. (New) The composition of claim 581, wherein the particulate apatite has an average crystal size of less than 100 nm.

586. (New) The composition of claim 581, wherein the particulate apatite is densified.

587. (New) The composition of claim 581, wherein the particulate apatite undergoes apatite phase decomposition of less than 10% when exposed to conditions of at least 1300 °C for at least 2 hours.

588. (New) The composition of claim 581, further comprising an auxiliary structural additive.

589. (New) The composition of claim 588, wherein the auxiliary structural additive comprises a ceramic additive.

590. (New) The composition of claim 589, wherein the ceramic additive comprises a metal oxide.

591. (New) The composition of claim 590, wherein the metal oxide comprises zirconia.

592. (New) The composition of claim 588, wherein the auxiliary structural additive is a metal or alloy.

593. (New) The composition of claim 588, wherein the auxiliary structural additive is present in an amount of between about 1% and about 50% by volume.

594. (New) The composition of claim 581, wherein the particulate apatite is carbonated apatite.

595. (New) The composition of claim 581, wherein the particulate apatite comprises an organic species.

596. (New) The composition of claim 595, wherein the organic species is a self-assembling surfactant or a polymer.

597. (New) An article comprising a densified apatite structure having a dimension of at least about 0.5 cm and an average XRD crystal size of less than about 250 nm, wherein the apatite structure undergoes phase decomposition of less than about 10% when exposed to conditions of at least about 1300 °C for at least about 2 hours.

598. (New) The article of claim 597, wherein the densified apatite structure has a density of at least about 90%.

599. (New) The article of claim 598, wherein the densified apatite structure has a density of at least about 98%.

600. (New) The article of claim 597, wherein the densified apatite structure has a porosity of greater than about 20%.

601. (New) The article of claim 597, wherein the densified apatite structure has a compressive strength of at least about 150 MPa.

602. (New) The article of claim 601, wherein the densified apatite structure has a compressive strength of at least about 500 MPa.

603. (New) The article of claim 597, wherein the phase decomposition is less than about 5%.

604. (New) The article of claim 597, wherein the densified apatite structure has an average XRD crystal size of less than about 150 nm.

605. (New) The article of claim 597, wherein the densified apatite structure comprises an auxiliary structural additive.

606. (New) The article of claim 605, wherein the auxiliary structural additive comprises a ceramic additive.

607. (New) The article of claim 606, wherein the ceramic additive comprises a metal oxide.

608. (New) The article of claim 607, wherein the metal oxide comprises zirconia.

609. (New) The article of claim 605, wherein the auxiliary structural additive is nanocrystalline.

610. (New) The article of claim 605, wherein the auxiliary structural additive is a metal or alloy.

611. (New) The article of claim 605, wherein the auxiliary structural additive is present in an amount of between about 1% and about 50% by volume.

612. (New) The article of claim 597 wherein the densified apatite structure comprises carbonated apatite.

613. (New) The article of claim 597 wherein the densified apatite structure comprises an organic species.

614. (New) The article of claim 597 wherein the article is at least part of a prothesis.

615. (New) The article of claim 614, wherein the article is a prothesis.

616. (New) The article of claim 597 wherein the article comprises an exterior coating on a prosthesis.

617. (New) The article of claim 597 wherein the article is a bioactive implant.

618. (New) The article of claim 617, wherein the bioactive implant is an orthopedic or dental implant.

619. (New) The article of claim 612, wherein the carbonated apatite is a reactive layer on a bioceramic capable of enhancing bioactivity for bone growth.

620. (New) An article comprising an apatite structure having a dimension of at least about 0.5 cm, a density of at least about 98%, and an average XRD crystal size of less than about 250 nm.

621. (New) The article of claim 620, wherein the apatite structure undergoes phase decomposition of less than about 10% when exposed to conditions of at least about 1300 °C for at least about 2 hours.

622. (New) The article of claim 620, wherein the apatite structure has a compressive strength of at least about 150 MPa.

623. (New) The article of claim 620, wherein the apatite structure has a compressive strength of at least about 500 MPa.

624. (New) The article of claim 621, wherein the phase decomposition is less than about 5%.

625. (New) The article of claim 620, wherein the apatite structure has an average XRD crystal size of less than about 150 nm.

626. (New) The article of claim 620, wherein the article is a prothesis.

627. (New) The article of claim 620, wherein the article is at least part of a prothesis.

628. (New) The article of claim 620, wherein the article comprises an exterior coating on a prosthesis.

629. (New) The article of claim 620, wherein the article is a bioactive implant.

630. (New) The article of claim 629, wherein the bioactive implant is an orthopedic or dental implant.

631. (New) The article of claim 620, wherein the apatite structure comprises an auxiliary structural additive.

632. (New) The article of claim 631, wherein the auxiliary structural additive comprises a ceramic additive.

633. (New) The article of claim 632, wherein the ceramic additive comprises a metal oxide.

634. (New) The article of claim 633, wherein the metal oxide comprises zirconia.

635. (New) The article of claim 631, wherein the auxiliary structural additive is nanocrystalline.

636. (New) The article of claim 631, wherein the auxiliary structural additive is a metal or alloy.

637. (New) The article of claim 631, wherein the auxiliary structural additive is added in an amount of between about 1% and about 50% by volume.

638. (New) The article of claim 620, wherein the apatite structure comprises carbonated apatite.

639. (New) The article of claim 638, wherein the carbonated apatite is a reactive layer on a bioceramic capable of enhancing bioactivity for bone growth.

640. (New) A method for preparing particulate apatite comprising:

- (i) precipitating apatite by mixing a calcium source and a phosphate source, wherein the pH of the mixture is about 7 to about 14,
- (ii) aging the precipitated apatite,
- (iii) controlling the conditions of steps (i) and (ii) so as to produce an apatite precipitate having an average crystal size of 150 nm or less,
- (iv) collecting the apatite precipitate, and
- (v) drying the apatite precipitate to obtain a particulate apatite having an average crystal size of 150 nm or less and a particle size of 1  $\mu$ m or less.

641. (New) The method of claim 640, wherein the calcium source has a concentration of less than about 1 M.

642. (New) The method of claim 641, wherein the calcium source has a concentration of less than about 0.5 M.

643. (New) The method of claim 640, wherein the calcium source is calcium nitrate and the phosphorus source is diammonium hydrogen phosphate.

644. (New) The method of claim 640, wherein the calcium source and phosphate source has an atomic ratio (Ca/P) of about 1.67.

645. (New) The method of claim 640, wherein the calcium source is added to the phosphate source.

646. (New) The method of claim 645, wherein the calcium source is added to the phosphate source at an addition rate of less than about 0.010 mole per minute.

647. (New) The method of claim 640, wherein the mixture has a pH of between about 11 and about 13.

648. (New) The method of claim 640, wherein the precipitated apatite is aged at a temperature of about -25 °C to about 100 °C.

649. (New) The method of claim 648, wherein the precipitated apatite is aged at a temperature of about 20°C.

650. (New) The method of claim 640, wherein the precipitated apatite is aged for at least 12 hours.

651. (New) The method of claim 640, further comprising grinding the precipitated apatite.

652. (New) The method of claim 651, wherein the grinding step comprises wet grinding.

653. (New) The method of claim 640, wherein the step of precipitating comprises adding an auxiliary structural additive.

654. (New) The method of claim 653, wherein the auxiliary structural additive comprises a ceramic additive.

655. (New) The method of claim 654, wherein the ceramic additive is a metal oxide.

656. (New) The method of claim 653, wherein the auxiliary structural additive is a metal or alloy.

657. (New) The method of claim 640, wherein the step of precipitating comprises adding a solution of an organic species.

658. (New) The method of claim 657, wherein the organic species is a self-assembling surfactant.

659. (New) The method of claim 658, wherein the organic species is a polymer.

660. (New) The method of claim 640, wherein the step of precipitating comprises adding a carbonate source.

661. (New) A method of producing a densified apatite structure comprising:

(i) providing particulate apatite having an average particle size of 1  $\mu\text{m}$  or less, an average crystal size of 150 nm or less, and a surface area of at least about 40  $\text{m}^2/\text{g}$ , and

(ii) densifying the particulate apatite at a temperature of no more than 1100 °C to produce a densified apatite structure having a dimension of at least about 0.5 cm and an average XRD crystal size of less than 250 nm.

662. (New) The method of claim 661, wherein the particulate apatite is densified by sintering.

663. (New) The method of claim 662, wherein the particulate apatite is densified by pressureless sintering.

664. (New) The method of claim 662, wherein the particulate apatite is densified by pressure-assisted sintering.

665. (New) The method of claim 661, wherein the particulate apatite is densified at a temperature of no more than 1000 °C.

666. (New) The method of claim 661, wherein the densifying step occurs in the absence of sintering aids.

667. (New) The method of claim 661, wherein the particulate apatite is densified by colloidal pressing.

668. (New) The method of claim 661, further comprising the step of calcining the composition at a temperature of less than about 1000 °C.

669. (New) The method of claim 661, wherein the densifying step occurs for a period of time of no more than about 2 hours.

670. (New) The method of claim 661, further comprising allowing phase decomposition of less than about 10 % in the article.

671. (New) The method of claim 661, wherein the particulate apatite comprises an auxiliary structural additive.

672. (New) The method of claim 661, wherein the particulate apatite comprises carbonate.

673. (New) The method of claim 661, wherein the particulate apatite comprises an organic species.

#### REMARKS

The above claims are identical to claims 581-673 filed in the preliminary amendment of May 31, 2002, except that these claims have been underlined as requested by the Patent Office.

**CONCLUSION**

If, for any reason, the Examiner is of the opinion that a telephone conversation with Applicants' representative would expedite prosecution, the Examiner is kindly invited to contact the undersigned at (617) 720-3500.

Please charge any fee or fee deficiency occasioned by this amendment to Deposit Account No. 23/2825.

Respectfully submitted,

  
\_\_\_\_\_  
Timothy J. Oyer, Reg. No. 36,628  
Wolf, Greenfield & Sacks, P.C.  
600 Atlantic Avenue  
Boston, Massachusetts 02210-2211  
Tel. No.: (617) 720-3500  
Fax No.: (617) 720-2441

Docket No. M00925/70110

Date: May 5, 2002